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International application number: PCT/US05/007925

International filing date: 08 March 2005 (08.03.2005)

Document type: Certified copy of priority document

Document details: Country/Office: US  
Number: 60/606,985  
Filing date: 03 September 2004 (03.09.2004)

Date of receipt at the International Bureau: 18 April 2005 (18.04.2005)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)



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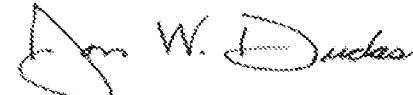
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APPLICATION NUMBER: 60/606,985

FILING DATE: *September 03, 2004*

RELATED PCT APPLICATION NUMBER: PCT/US05/07925

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090304

17712 U.S. PTO

# HAMILTON, BROOK, SMITH & REYNOLDS, P.C.

## PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 C.F.R. 1.53(c)

00746 U.S.PTO  
60/606985

090304

Express Mail Label Number EL 955640325 US

Docket Number 0003.2004-000

### INVENTOR(S)

Given Name (first and middle (if any))	Family Name or Surname	Residence (Street Address, city and either state or Foreign Country)
Norwin W.	Wolff	

Additional inventors are being named on the separately numbered sheet(s) attached hereto

### TITLE OF THE INVENTION (500 characters max)

Water-Based Olefin/Styrene/Acrylic Polymer System for Multi-Functional Cosmetic Applications

### CORRESPONDENCE ADDRESS

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### ENCLOSED APPLICATION PARTS (check all that apply)

<input checked="" type="checkbox"/> Specification Number of Pages [ 8 ]	<input type="checkbox"/> Other (specify) _____
<input type="checkbox"/> Drawing(s) Number of Sheets [ ]	_____

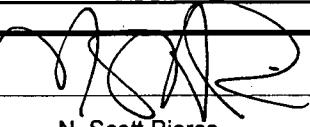
### METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT (check one)

<input checked="" type="checkbox"/> A check or money order is enclosed to cover the filing fees. Please charge any deficiency in fees and credit any overpayment to Deposit Account Number 08-0380.	FILING FEE AMOUNT (\$)
<input type="checkbox"/> The Director is hereby authorized to charge filing fees or credit any overpayment to Deposit Account Number 08-0380.	\$160
<input type="checkbox"/> Previously Submitted.	

The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.

No

Yes, the name of the U.S. Government agency and the Government contract number are: \_\_\_\_\_

Signature		Date	9/3/04
Submitted by Typed or Printed Name	N. Scott Pierce	Reg. Number	34,900

**PROVISIONAL APPLICATION COVER SHEET**

***Additional Page***

September 3, 2004 Express Mail Label No. EL 955640325 US

## PRIVILEGED COMMUNICATION

Docket No. \_\_\_\_\_

Interpolymer Corporation

**INVENTION DISCLOSURE STATEMENT**

(See Instructions on Next Page.)

1. Inventor(s) Names (*actual inventorship to be established by patent counsel*):

Norwin W. Wolff, Timothy L. Martin, William Tenney, Joseph J. Cincotta

2. Descriptive title of the invention:

Water-based olefin / styrene / acrylic polymer system for multi-functional cosmetic applications

3. Description of the invention: (see instructions on next page)

The present invention relates to the unique properties of olefin/styrene/acrylic copolymers which can provide multifunctional properties in hair care products and other keratinous surfaces using a single vehicle. Polymers of the invention are prepared in hair fixatives, leave-in conditioners and shampoos and impart a combination of properties unique to these polymers. They exhibit excellent fixative properties with high humidity curl retention while the same polymer will reduce the washout of color from color-treated hair. Due to the elasticity of the polymer under mild heat conditions, the same polymer, with the use of a hair dryer, will impart renewable styling to hair without additional fixative application.

Polymers of this invention are in emulsion form with emulsions have a molecular weight in the range of 2000 - 1,000,000. Particle size of the emulsion particles range from 10 to 250 nm. The polymers are film formers with a high degree of elasticity from 50 to 500%. Minimum Film Formation Temperatures (MFT) range from 0 to 352<sup>5</sup>°C.

The emulsions of these polymers may be anionic, non-ionic, cationic or amphoteric.

Read, Understood and Witnessed by:

Inventor(s) Signatures:

Date

Witness 1

Date

Date

Witness 2

Date

Date

Date

<p>6. Was this invention made on: (Give details)</p> <p>- A Government Contract      ~ [Interpolymer] Funded Project      ~ A Proposal Effort      ~ Other</p> <p><b>Interpolymer R&amp;D funded project</b></p>				
<p>7. Briefly describe any actual reduction to practice or experimental work which has already been done or which is planned and include references to log books or other supporting documents, models, samples, photographs, test data or computer records: High humidity curl retention studies vs competitive products, color retention studies using color spectrophotometer, dry hair evaluations per industry standards have all been performed.</p>				
<p>8. State the commercial uses for this invention: Cosmetic Products</p>				
<p>9. List references or prior art (prior patents, printed publications, products, works of others) known to you:</p>				

Inventor(s) Signatures:

Read, Understood and Witnessed by:

Date	Witness 1	Date
Date	Witness 2	Date
Date		
Date		

**INSTRUCTIONS**

Please complete and submit [to the (Company Name) Patent Committee] an Invention Disclosure Statement for each invention you make. This statement is submitted for the purpose of obtaining legal advice regarding the patentability of your invention. Do not attempt to decide on your own whether your invention is patentable. This determination will be made by [the Company Name] Patent Counsel. This form is an important privileged legal document; it should be legible and as complete as possible. In completing the description of the invention, please provide the following: problem solved by your invention; how your invention solves the problem; advantages over other methods to solve the problem; results achieved; features believed to be novel; and any sketches which help illustrate the invention. Additional sheets can be used, but each must be attached to this form and each must be signed and witnessed. Any additional information not requested which you feel would be helpful [to the [Company Name] Patent Committee] in deciding whether a patent application should be filed should also be provided along with this form.

After you have completed this form, please forward the original signed and witnessed copy [to the [Company Name] Patent Committee] so it can be forwarded to Patent Counsel. Retain one copy for your files and submit a second copy to your immediate supervisor. Do not further distribute this completed form.

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## Achieving Color Retention Properties in Hair Care Products Using Novel Grafted Polymers.

### I. Introduction:

The desire to change the color of one's hair has existed since ancient times. Greeks and Romans used everything from harsh soaps to boiled walnuts to modify hair color in order to meet the fashions and ideologies of the day. Today, modern chemistries have provided a much wider spectrum of colors with significant improvements in hair aesthetics and permanency. With the many advances made in hair treatment formulas, the current challenge faced by many formulators is how to improve the longevity of color treatment without the loss of color components from the original color treatment. This paper addresses another possible way to improve the permanency of hair treated with a synthetic (oxidative) color.

### II. Olefin-Graft Polymer Technology:

Acrylic based polymers have been manufactured with a variety of functional monomers and side chains to offer diverse features and benefits to the formulator. Polyolefins, from parrafin waxes to polyethylene derivatives, have similarly provided a variety of benefits over the years. The grafting of these two diverse compounds for the personal care market offers some interesting formulating properties. Polymers of this type have been used in the industrial market for almost three decades bringing improved properties to a variety of surfaces. However, by proper selection of olefin backbone and acrylic side chain, functional groups can be attached to provide properties for the personal care formulator not easily achieved by an individual component.

The olefin is semi-crystalline in nature and can provide features such as barrier and moisture retention, a reversible heat-activated elasticity and lubricity. This portion we designate as the Crystalline Polymer Portion or CPP. The acrylic side chains of this particular composition are soft, non-tacky and in all but one case contain amino functionality. For ease of nomenclature, we call this the Amorphous Polymer Portion or APP. The APP contributes better film formation, adhesion, substantivity and stability to the olefin.

(Figure 1: insert picture of floating wax particles)

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*The lower density of an olefin would normally force its migration to the surface of a film during drying. By utilizing the grafting process, the APP portion of the polymer has made the CPP or olefin portion denser and thus more evenly distributed throughout the film.*

One possible structure for polymers of this type involves the bonding and entangling of styrene or acrylic polymers within the olefin polymeric layers, much like a sandwich. The simple depiction below (Fig. 2) is of a graft polymer showing how the different segments of the CPP and the APP could extend along different planes. This is assisted by the acrylic chains' higher degree of flexibility than that of the semi-crystalline olefin. Depending on where this grafting interaction occurs, the olefin section will affect the conformation of the styrene/acrylic portion. By proper control of the hydrophilic/hydrophobic balance, it is possible to obtain a film with good acrylic, substantive properties while maintaining the olefin's barrier properties.

(Figure 2: insert picture of olefin graft molecule)

### III. Color Treatment/Mechanism of Action:

Hair coloring products work in different ways depending upon the level of coloring desired. These have been described by the industry as Level 1, semi-permanent color; Level 2, demi-permanent color and Level 3, permanent color. Each increasing level involves the additional use of chemicals to alter the color composition of the hair.

Level 1 colors do not appear to dramatically affect the natural color of the hair since the color molecules that enter the cuticle do not interact with the hair's natural pigment.

Level 2 colors appear to use a small amount of peroxide in order to enhance color but, unlike Level 3, they do not contain ammonia. During dyeing the Level 2 pre-color molecules penetrate the cuticle and enter the cortex where they partner to create medium sized molecules. These larger size molecules retard or prevent the degradation process of color fading.

Finally, Level 3 colors use both ammonia and peroxide to lighten the hair's natural pigment, in addition to adding a new permanent color. The ammonia can cause the cuticle to swell allowing the color precursor to enter the cortex. The precursors react and expand to a size to which they can not easily be washed out. The final color is a combination of the natural color and the new shade chosen.

For this study, we chose to use Level 2 colors which provided more dramatic and easily measurable distinctions between wash cycles. If the tested products in question performed in limiting dye loss during washing with Level 2 colorants, then we felt confident that these results would also translate well for Level 3 colors. The color loss of

Level 2 colorants during hair washing cycles can be partially attributed to anionic and amphoteric shampoo ingredients which can draw oppositely charged dye molecules from the hair cortex through the openings in the hair cortex. This gradual leaching of the color molecules through the openings in the cuticle is the primary cause of color loss at this level. UV degradation is another means of color loss and is not being addressed by this process but can be mollified by the use of UV absorbers which are known to the industry. This is a particular problem with Level 3 colors.

Block polymers, silicones and other assorted compounds have been used by formulators in an effort to retard the leaching of colorants and provide a protective barrier. These barriers can either function by occlusion or by forming an ionic barrier to protect the color in the hair shaft. Olefin graft polymers appear to work as occlusive barriers but contain both anionic and cationic charges which can serve to limit shampoo interaction with most dye molecules. As referenced above, olefin graft polymers can provide substantivity to the hair shaft via the acrylic portion and use the crystalline olefin portion to occlude the cuticle openings limiting color loss.

### III. Color Retention Testing Protocol:

#### Material List:

- Bleached blonde human hair was obtained from International Hair Importers and Products (IHIP) of White Plains, NY. The hair was swatched by Interpolymer Corporation and secured with wound wire and rubber cement. Each tress was three (3) grams in net weight before tressing and was six inches in length.
- A Level 2 commercial oxidative dye (Dark Brown) was used to treat the hair.
- The shampoo chosen was a commercial shampoo for normal hair with no color retention claims.
- A laboratory prepared leave-in conditioner using an olefin graft polymer was used. (see below formula)
- A laboratory prepared control leave-in conditioner without a graft polymer was used.

Figure 3

#### LEAVE-IN CONDITIONER WITH 5% OLEFIN GRAFT POLYMER

	<u>Weight %</u>
Phase A	
Distilled Water	87.93
Disodium EDTA	0.05
Propylene Glycol	1.50
Panthenol	0.30
PG-Hydroxyethylcellulose Cocodimonium Chloride	2.50
Cetrimonium Chloride	0.75
Polyquaternium-59 (and) butylenes glycol	1.00
Phase B	
DMDDM Hydantoin	0.40

Hydroxypropyltrimonium Hydrolyzed Silk	0.10
Hydrolyzed Silk	0.05
Phase C @45°C	
Oleth-20	0.35
Fragrance	0.07
Phase D	
<b>Ethylene/Styrene/Acrylates Copolymer (pending)</b>	5.00
Citric Acid	Q.S.
YIELD:	100.00%

Procedure

- Warm water to 35°C. Add each ingredient as listed. Stir between each addition until clear, homogenous solution results. Add Phase C at 45°C. Stir until clear. Add Phase D. Adjust pH.

Initial pH 6.29. Add approximately 0.10g citric acid to drop pH to 4.65.



## Procedure:

Each tress was washed and rinsed before treating with the color to eliminate any contamination from the tress preparation. A minimum of 5.5 grams of the hair color was applied to the hair tress and spread evenly and thoroughly on both sides of the tress using a wide coloring brush and fingers. The tresses were allowed to process on aluminum foil for the manufacturer's recommended time at ambient temperature, approximately 23 degrees C. The tresses were then rinsed with running tepid water for two minutes. The tresses were then allowed to dry for a minimum of two days before testing began.

## Treatment Cycle:

1. Shampoo: Wet tress and apply two (2) grams of shampoo. Spread the shampoo evenly through the tress and then massage between the fingers from top of tress to bottom five times to create a lather. Rinse with tepid water until free of soap, approximately 15 seconds. Squeeze out the excess water using the middle and index finger and proceed.
2. Conditioner: Apply two grams of conditioner formula (test or control) is applied to the wet tress and allowed to rest for one minute. Comb the tress with a wide tooth comb to remove excess conditioner and then dried.
3. Drying: Oven dry the swatches for one hour at 35 C.

The above cycle was repeated Eight (8) times, with measurements taken at intervals of 2, 5 and 8. The hair samples were then evaluated for color loss using a Series Sphere Spectrophotometer, X-Rite Model SP-62. L\*, a\*, and b\* values are measurements of color on a 3-D grid as illustrated in (Fig. 3) the chart below. Finally color retention, expressed as a percent, was calculated by determining the Delta E value representing the color shift along the three color coordinates after each treatment interval. Delta E values were correlated using an undyed hair standard and dyed hair with no treatment standard.

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(Figure 3: L\* a\* b\* charts)

Initial results showed approximately 15% improvement in color retention with the olefin graft conditioner over the control conditioner over a period of 8 wash and condition cycles. These results proved interesting and additional work was performed.

(Figure 4: insert bar chart with results from first round of tests)

A second round of tests were conducted in an effort to increase the ability to extend the period-of-use claimed and/or the percent of color retention. Modifications were made to the testing protocol as follows:

1. In order to benchmark the performance of the olefin graft polymer systems, the test was modified to use a commercially available, mild, baby shampoo for all of the swatch washings, with the exception of one set.
2. A set of tresses was, instead, washed with a commercially available shampoo claiming 45% color retention properties in a Level 3 application.
3. The color treatment was changed from Level 2 brown to Level 2 Intense Auburn.
4. The complete testing protocol included:
  - a. Tresses washed with baby shampoo and treated with control conditioner;
  - b. Tresses washed with baby shampoo and treated with Ethylene/Acrylate Copolymer (and) Acrylates Amino Methacrylate Copolymer (pending) olefin graft conditioner;
  - c. Tresses washed with baby shampoo and treated with the previous olefin graft conditioner;
  - d. Tresses washed with the color retentive shampoo and treated with the control conditioner.

The drying cycle was also modified. After washing and treating with the conditioners, the swatches were combed with a wide toothcomb while being dried with a hair dryer on maximum setting. This was done for three (3) combings to eliminate excess conditioner and water. The swatches were then oven dried for 25 minutes at 40° C. If any residual moisture was evident at the end, then the hair dryer was used to finish moisture removal. The above cycle was repeated 12 times, with measurements taken at intervals of 4, 8, and 12. The hair samples were then evaluated for color loss using the same procedure as outlined above using the spectrophotometer. The resultant L\*, a\*, b\*, values were measured and the Delta E calculated. A chart demonstrating the percent color retention is found in figure 5.

(Figure 5: insert bar chart with results from modified tests)

As one can tell, there was a significant increase in color retention with the revised test and protocol. It was important for us to notice the improved characteristics of the amino-modified olefin graft in comparison with the previously tested olefin graft. One of the observations made during the test concerned an increased combing effort on the olefin

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graft conditioners compared to the standard. We were concerned that damage to the hair or the protective barrier of the olefin graft was causing the drop in color retention as washings increased. In order to verify this, a third study was conducted in which the conditioning formula was modified to double the cetrimonium chloride while the olefin, Ethylene /Acrylate Copolymer (and) Acrylates Amino Methacrylate Copolymer, was cut to 1.5%. This was again tested against the commercial color retentive shampoo of the second study using the dark brown Level 2 color. The results of this study are found in figure 6.

(Figure 6: insert bar chart results from test)

#### **Application Flexibility:**

In order to understand the potential effects of olefin polymers on hair color formulations, another method of incorporation was evaluated. To check the effects of the olefin polymer in the dye preparation, we prepared a commercial Level 3 hair dye preparation per the manufacturer's instructions. This dye preparation was split into equal parts. The Ethylene /Acrylates Copolymer (and) Acrylates Amino Methacrylate Copolymer was added to one of the preparations at a 2% active level. The two dye preparations were then used to treat similar hair swatches per the manufacturer's instructions. These swatches were also measured for initial color and subjected to the same wash cycles using the commercial baby shampoo. This test indicates the amino modified olefin graft polymer can help retain color if the application is in the coloring process. The results of the test are shown in figure 7. This method did not incorporate the use of a conditioner or other color-retention aid.

(Figure 7: insert results from final test)

#### **IV. Results and Conclusions:**

The overall positive results generated by these studies indicate that the olefin graft polymers increase color retention of synthetic, oxidative dyes. The degree of performance will be determined by formulation, application and percent of polymer solids. New technologies (such as olefin-graft variations) can assist formulators in finding the right balance of substantivity and removability while maintaining or enhancing the appropriate aesthetic qualities desired in hair coloring and maintenance systems.